Preparation and Testing of Ice-Enriched Lunar Regolith Simulant for RESOLVE OVEN. Bonnie Cooper[1], Aaron Paz[2], and Matt Smith[3], [1]Jacobs ESCG, P.O. Box 58447, JE23, Houston TX 77258-8447, bonnie.l.cooper@nasa.gov), [2]NASA Lyndon B. Johnson Space Center, [3]East Tennessee State University.

Introduction: The RESOLVE (Regolith and Environment Science and Oxygen and Lunar Volatile Extraction) project seeks to deploy a robotic ISRU lander at the lunar south pole to determine the accessibility of frozen ice and the resource potential at various locations. The OVEN (Oxygen and Volatiles Extraction Node) component will be used to heat samples to 150°C or higher in order to drive off ice and other volatiles, which will then be identified and measured.

An ice volatilization system has been developed to specifically collect data on the extraction of water vapor from a sample. Testing of the system was begun in April of this year. This report addresses our current work to test the system in a relevant environment—in this case, using simulants with up to 10wt% water ice at 76K, in reduced atmosphere.

Simulant Preparation Techniques:

Freezer: A walk-in freezer at NASA Johnson Space Center's Astromaterials Research and Exploration Sciences (ARES) directorate was used for sample preparaton. This facility allowed us to place all necessary equipment within a space that maintained a temperature at or below -10°C. In order to prevent condensation of ambient humidity onto the samples during weighing, it was useful to weigh the samples inside the freezer.

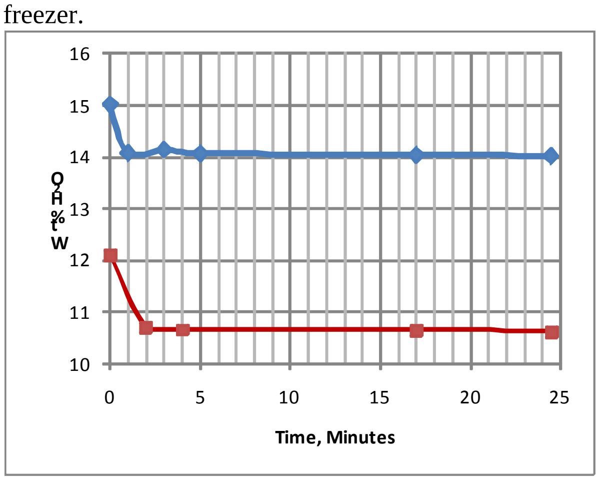

Figure 1. Wt% water retained after samples containing adsorbed water were placed in a -10°C freezer in air-tight containers. Blue = 15wt% starting H_2O content; red = 12wt% starting H_2O content.

Vapor method: In this method, a measured quantity of lunar simulant was placed in a sieve with a pore size of 30 µm. Water was placed in a sieve pan, and the sieve containing the soil was placed on the pan. This assembly was gently heated to allow vapor to adsorb onto the particles. The sieve containing the sample was weighed periodically to determine when the desired weight percent of water had been added to the sample. The sample was then placed in an air-tight container and transferred to the freezer.

Some of the ice sublimated away from the particles, in spite of being kept in an airtight container (Figure 1). The sample was weighed periodically to determine the rate of sublimation. It was learned that about 1.5wt% additional water should be added initially in order to produce a frozen sample with the desired weight percent of water.

Shaved ice method: In this method, small particles of ice are created with an off-the-shelf ice shaving machine. The ice and the shaver are located inside the walk-in freezer, to prevent incipient melting which could later re-freeze and weld the particles together. The ice shavings are mixed with dry frozen soil in the proper ratio. The mixture is placed inside a soil sieve (diameter 7.5 cm and pore size 30 µm). The sample mixture and the test fixture sample holder are bathed in liquid nitrogen, then the sample is reweighed and placed in the sample holder. The test begins as soon as the wiring is connected (about 10 minutes is required).

Figure 2. Test fixture. The heating chamber has a sample volume of approximately 7.62cm diameter and 1.27 cm height.

Hardware Configuration: The ice volatilization system is designed to gather several critical data points pertinent to the functionality of the RESOLVE OVEN. The first objective is to verify that all water placed in the heated vessel can be effectively removed and quantified. The second objective is to observe a detailed heating profile for the sample. The temperature gradient across the sample, heat input, heatup time, and pressure buildup are all critical data points for the RESOLVE OVEN. The system has three heaters. The one at the bottom of the sample chamber is used to heat the sample; the other two heaters are used to heat the tubing and fittings through which the water vapor flows, in order to reduce the amount of condensation that occurs inside the fixture. Heater #2 is a rope heater on the upper flange, cemented in place with Resbond920. Heater #3 is the same type of rope heater, and its purpose is to heat fittings and components from the quick disconnect to the valve.

Five thermocouples measure the temperature at various locations within the sample. Because heat comes primarily from the bottom, the thermocouples are placed at varying depths from the base of the heating chamber: 0.19mm, 0.36 mm, 1.37 mm, 2.44 mm, and 2.92 mm.

Testing: Tests run so far have shown that the ice volatilization system (Figure 2) can heat the samples evenly and extract the H_2O that is either adsorbed to the particles or mixed in as ice chips. One example test is illustrated in Figure 3. In this test, dry-frozen soil was mixed with ice particles, then the mixture was bathed in liquid nitrogen to reduce its temperature to approximately 76K. The total sample mass was 12.72 grams, with 14.5wt% H_2O (total H_2O mass of 1.84 grams). After the test, the sample weight was 10.86 grams. Within the error range of the measurements, all of the surface-correlated and crystalline H_2O was removed from the sample. Nevertheless, refinements are needed to ensure that condensation does not occur within the heater itself, so that all the water removed from the sample can be measured in the product container (in this case, a dessicant container).

Conclusions: Progress is being made in developing the RESOLVE system, and eliminating risk for future development of flight-qualified hardware. This report describes the current activities related to the ice volatilization vessel, which is designed to heat frozen regolith in order to remove adsorbed H_2O. Testing has shown that the ice volatilization system is capable of removing frozen ice and surface-adsorbed H_2O from lunar regolith simulant.

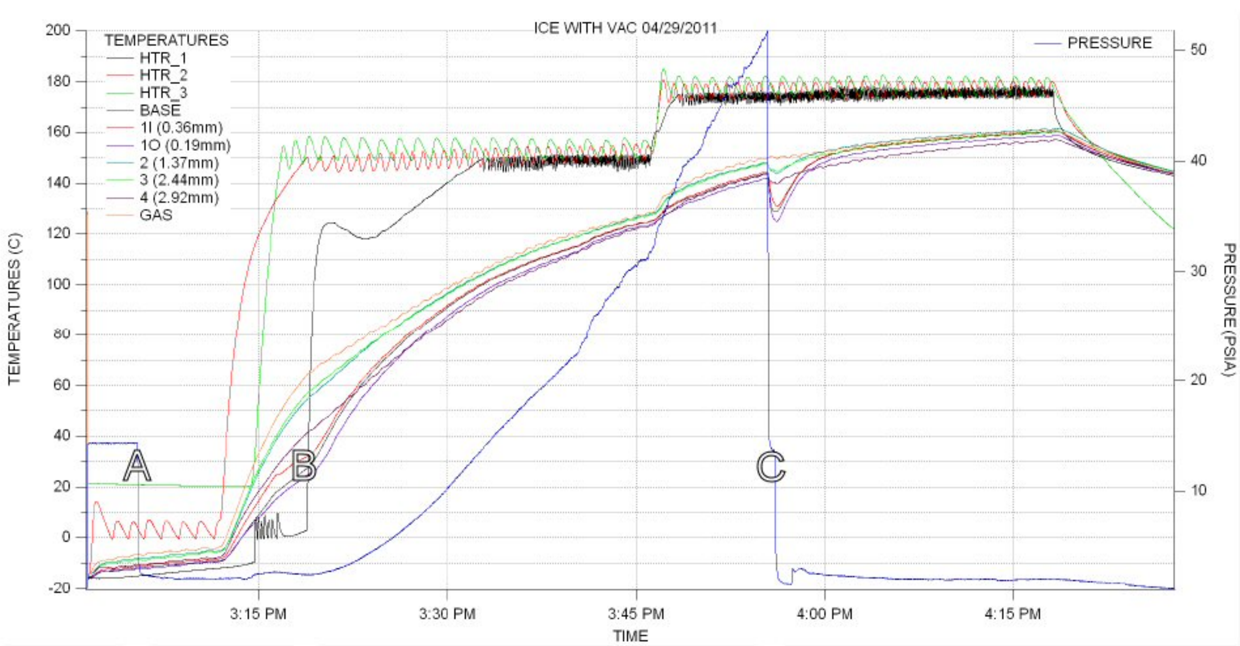

Figure 3. Test data for a sample cooled to 76K which included ice chips and lunar soil simulant. Point A: Started vacuum. B: Set heater to 150°C. C: Vented to vacuum.